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MONTHLY PROGRESS REPORT

No. 5

for

November - 1962

Submitted by

NEW PRODUCT RESEARCH of TAPCO

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Monthly Progress Report No. 5
December 14, 1962

I INTRODUCTION

This document represents a fifth monthly report covering the work on the experimental program for development of an "Osmotic Still" and improvements in the performance characteristics of the Ionics Dual Membrane Fuel Cell during the month of November, 1962. This development work is being accomplished under NASA-Lewis Research Center Contract No. NAS 3-2551 by the New Product Research Department of Tapco and Ionics, Inc. as a subcontractor to Tapco.

II OVERALL PROGRESS

A. Tapco Portion of Program

1. A total of four tests with American Machinery and Foundry cation membranes were made. In each of these tests extracted water in condenser was contaminated with acid. Also, in each case post test examination of membranes and gaskets revealed them to be sound and without any defects.

2. Above tests led to suspicion that there might be an unknown phenomena occurring during the tests that produced acid contamination in the extracted water. As a result, a single AMF cation membrane was tested with one side exposed to the air while the other to electrolyte (sulphuric acid) of usual concentration (30 percent). This test revealed acid sweating of membrane on the air side as soon as electrolyte started circulating on the opposite side. This sweating occurred only during the initial stages of testing and probably was the cause of acid contamination in our previous four tests. More details on this test are given in Section IV.

3. A test procedure to eliminate this initial acid sweating was worked out.

4. Further tests were made with AMF anion membranes utilizing procedures worked out in part (3) above. These tests were successful thus substantiating our suspicions described under part (2) above. More details on this test are given in Section IV.

5. An experiment to coat the Monel metal support screens with a thin layer of Teflon was performed in order to obtain non-wetting screen surface and also to provide an acid resistant coating on the Monel metal screen used as filler in the test housing as shown in Figure 2 of Monthly Progress Report No. 1. These coated screens were used in the successful tests described under part (4) above.

6. Tests to date indicate that AMF membranes are superior in this application to other membranes tested. The AMF membranes can withstand 200°F operation, 15 psi pressure drop across the membrane and they are extremely durable compared to the Ionics membranes.

B. Ionics Portion of Program

1. Two cells were placed on life test at 30°C using the gas and electrolyte compartments machined from pieces of 60 mil epoxy glass in place of the compartments laminated from 15 and 30 mil stock.

B. Ionics Portion of Program Cont'd

2. A constant temperature bath capable of holding four cells at 60°C has been completed. Operation of two cells in this bath was initiated on December 4.

3. Duplicate samples of gold plated titanium, Trilok mesh, epoxy, buna N, plaskon, Kel-F, 9 oz. glass backed membranes and monel metal have been immersed in 6N H_2SO_4 and the bath maintained at 60 and 95°C.

4. The Clevite Corporation in Cleveland, Ohio, has been asked to provide Ionics with nine samples of the sintered type electrodes.

5. Paste and sintered electrodes have been prepared for the 60°C runs.

6. Control equipment for four 60°C cells has been set up. A 24 point recorder has been set up to provide a permanent record of voltages.

III CURRENT PROBLEMS

A. Tapco Portion of Program

The problem of acid contamination of the water condensate is believed to be overcome. It is felt, however, that the solution of this problem was partly due to the non-wetting material used to support the membrane. The Teflon coating used to cover Monel metal screens was not satisfactory and, therefore, further work on techniques of coating with Teflon and Kel-F materials is needed. It should be noted that coating of Monel metal screens will serve two purposes. One, to produce non-wetting surface and second, to protect metal screen against attack by sulphuric acid.

B. Ionics Portion of Program

1. Production of uniform sintered electrodes.

2. The elimination of small air pockets at top of cell. These can be instrumental in producing desiccation of membranes in conjunction with the incoming hydrogen and oxygen gases which are essentially vented toward the top of the membrane.

3. Resistance of materials to 6N H_2SO_4 at elevated temperatures.

IV NEXT MONTH'S EFFORTS

A. Tapco Portion of Program

1. Further Teflon and Kel-F coating techniques to cover metal screens supporting membranes will be conducted.

2. Performance tests will be continued with AMF anion and cation membranes.

B. Ionics Portion of Program

1. Continued material analysis at 60° and 95°C. It is hoped the proposed subcontract on materials evaluation can be approved.

B. Ionics Portion of Program

2. Testing of electrodes produced by Clevite Corporation on 2" x 2" test cells.

3. Life testing of 8-12 one-cell fuel cells at 60°C.

4. Preliminary design of a five-cell multiple cell configuration.

5. The introduction of measures to eliminate air pockets or their effects viz. the introduction of O_2 and H_2 at bottom of cell to avoid desiccation problems.

V TEST RESULTS

A. Tapco Portion of Program

1. The initial test was made with AMF cation membrane with 70°F sulphuric acid circulating through the back side while a vacuum was drawn slowly on the vapor side of membrane. Drops of liquid with high acid content accumulated in the condenser. This accumulation was thought to be from a membrane leak and the test unit was dismantled for inspection. No cracks or any other defects were found by visual inspection as well as with leak detector.

2. The second test with the AMF membranes was similar to the one described above. Again acid accumulated in the condenser and again there were no cracks and holes in membrane.

3. The third test was initiated with AMF cation membranes and was operated from room to 150°F electrolyte temperature. The increase in temperature of electrolyte, it was reasoned, will increase the water yield and will clean out the acid accumulation during the starting period. This test was operated for 13 hours over a period of three days with condenser water samples taken every hour. In this case, water of Ph 2 was obtained each time. Although no holes were found in the membrane, it was thought that the failure was possibly due to leakage of acid through the gasket.

4. An alternate gasketing arrangement was tried along with the introduction of a Marlex plastic screen as a membrane support material. This test also failed because water collected was of Ph 2 in spite of the fact that no cracks or holes were found in the AMF cation membranes used.

5. The reported failures of tests to obtain water with a Ph rating above 4 without evidence of membrane leakage lead to suspicion that an unknown membrane behavior was taking place that resulted in contamination of water with acid. As a result, a test was set up with an AMF cation membrane, such that a visual observation of membrane was possible during operation. This test revealed that upon introduction of acid at room temperature in the acid cavity of the test rig, the exposed face of membrane started to evolve liquid droplets of Ph 1. The evolution of droplets was excessive enough to cause vertical liquid runs on the membrane surface and collection of liquid at the bottom of a test unit. Increasing acid temperature produced a drying effect on the entire surface of the membrane. Once the surface was dry, the "sweating" could only be induced by the application of acid solution to the surface of membrane. This test indicated, therefore, that in all previous tests with the AMF membranes, the contamination of condensed water was due to the acid liquid evolved during the initial stages of tests, and which was carried by

A. Tapco Portion of Program Cont'd

5. the water vapor into the condenser and not by membrane or gasket leakage.

6. In order to protect the metal screen supports from acid attack and also to have a non-wetting material in contact with the membrane, a Teflon coating was applied to the Monel screens by dipping the screen in Teflon dispersion and sintering at 700°F for 30 seconds.

7. Another test with AMF anion membrane was initiated employing new assembly and "drying" procedures evolved after the tests described under Part (5) above were made. The membrane support screens in this test were coated by a procedure indicated under Part (6) above. The membranes were tested for approximately 19 hours at acid temperatures ranging from room temperature to 203°F and condensing temperatures ranging from room to 155°F. During this time of operation water extracted from sulphuric acid ranged from Ph 4 to Ph 5. This test provided our best results to date and was continued into the next reporting period.

B. Ionics Portion of Program

1. Control and regulatory systems at 30°C and 60°C have been completed.

2. Two duplicate test cells utilizing paste electrodes have been placed into bath for life-testing at 60°C. These cells performed well (see Table 1) but had to be removed due to failure of control system.

3. A compilation of life test data obtained up to the present is presented in Table 1.

4. Electroplated gold (50 microinches) did not withstand 6N H_2SO_4 at 95°C. Because of this and related material problems, life testing at 95°C has been discontinued until the materials problems have been explored in more detail.

VI QUALITY ASSURANCE

A. Tapco Portion of Program

The scheduled Quality Assurance and Engineering Review of progress at Ionics, Inc. was accomplished on November 15. In attendance at the meeting were representatives from the Quality Assurance and Engineering Departments of both NASA and TRW. A copy of Quality Assurance Trip Report is attached as Enclosure I to this monthly progress report. An initial earlier distribution of Enclosure I was made to the NASA and TRW personnel concerned.

A means for testing soundness of membrane material prior to use is still being pursued. Dupont makes a dye that is capable of pin-pointing porosity in a membrane while it is still protected by a water or Glycol solution. By application of the dye to one side of the moist membrane, it will seek out a void and define the area by a distinct visual show of color on the reverse side. A sample of this dye is on order but has not been received as of this date. Testing material with pre-determined leakage areas will be accomplished as soon as the dye is received. If this method proves successful it will be

much more economical and efficient than the previously suggested pressure test method because of the subsequent handling and fixturing problems pressure testing would impose.

An area of concern at this point in the program is the inability of our Purchasing Department to obtain proper physical and chemical analyses from the suppliers of membrane materials. There is a general reluctance in the plastics industry as a whole to supply positive information to the degree that repeat procurement of test qualified materials can be assured. This resistance to furnish requested certification, and pertinent technical performance data, with the material is based mostly on a desire to protect proprietary information. Unless this can be accomplished, reproducibility of product cannot be guaranteed.

B. Ionics Portion of Program

Quality assurance documentation was emphasized during the past reporting period.

Specifications have been revised and made current, incorporating the newly designed gas and electrolyte compartments. A Quality Assurance Manual for the Energy Conversion Department has been initiated and work is proceeding on developing quality control instructions for various critical steps in the processing and assembly of the fuel cells.

Ionics' Material Purchasing Specifications are to be initiated for those materials not having a nationally recognized standard, such as a NEMA or MIL specification.

Segregation of purchase material in an incoming inspection storage area is planned along with incoming inspection records. Inspected material will be identified and stored in separate applicable project areas.

Current plans are for the Quality Assurance Program to be fully operative at the institution of Task V or VI.

ENCLOSURE I

TRW QUALITY ASSURANCE TRIP REPORT ON VISIT TO IONICS, INC.

(Trip Made on November 15, 1962)

The purpose of the trip was to make a scheduled review of progress made at Ionics in accomplishing their planned Quality Control procedures to assure reproducibility of materials and effort in the NASA Fuel Cell Development Program.

Upon arrival at Ionics we proceeded directly to the area set aside for this project. We witnessed three cells that were undergoing immersion tests to check the effects of heat, at various degrees of temperature, on cell life and performance. At the time we observed the test, the water was at a temperature approximating ambient. The cells were partially submerged in the water with the electrical contacts at the electrodes exposed. Water temperature in the tank was controlled by a thermostatically operated heating unit. This particular test could not be considered as life cycling because of constant interruptions necessary to introduce changes and make various investigatjons as problems arose.

Ionics has assembled some working models with the change in configuration from their 6" x 6" standard type to the proposed 2" x 18". This change was made in order to increase the usable amount of the total surface area of the membranes. The square design had "DEAD" areas in the corners caused by lack of circulation of fluids and gases during operation. The new design reduces the inactive areas and permits functional use of more of the membrane surfaces by literally placing more of the area in line with the flow path of the gases and fluids. It is also felt that the long narrow design will be more adaptable to multiple cell construction and will fit more readily into any space made available in end use.

Additional design improvement changes, to reduce the number of sealing areas, are in progress. If successful these changes will introduce multiple related savings in material costs, reduced number of parts required, reduced amount of assembly time, eliminate leakage problems, and cut down on test rejections. The present problems and proposed corrections were demonstrated by one of the engineers who went through the actual build procedure for a single cell.

Ionics has made additional noticeable improvements in housekeeping techniques in the laboratory and test areas. Excess acid accumulations have been removed, to a satisfactory degree, from the life test units and attached control meters. The master meters have been calibrated by an independent laboratory working to National Bureau of Standard approved test masters. Certified results are on file at Ionics Quality Control Office.

Progress has been made in the Quality Control areas as defined by the nine points requiring attention in my call report of September 11 and 12. Internal control and handling of materials shows improvement. Ionics should be able to comply with NASA QA-1 at time of entry into Task V and VI as planned.

It should be mentioned, however, that in spite of efforts expended it is my feeling that Ionics is still faced with the same problems evident in September as far as unit construction and improved performance are concerned.

TABLE I

Cell #	Type of Electrode	Temp. °C.	Pressure, psig		Current Amperes	Average Voltage	Test Time (hrs)	Reasons for Disassembly
			Electrolyte	Gas				
9716	paste	30	20	15	4.0	0.72	12	Converted to cell #9718
9718	paste	30	20	15	4.0	0.78 0.64 0.66 0.72 0.70 0.60	12 192 288 408 552 696	Membrane drying due to air bubble at top of cell
9719	paste	30	20	15	4.0	0.59 0.60 0.51	20 192 264	Membrane drying due to air bubble at top of cell
9723	sintered	30	20	15	4.0	0.76 0.74 0.72 0.71 0.72 0.64	20 108 252 348 396 564+	Still running
9725	sintered	95	20	15	4.0	0.725	24	Component failure
9728	sintered	30	20	15	4.0	0.65 0.71 0.72 —	20 156 300 432	Membrane drying due to air bubble at top of cell
9734a	paste	60	20	15	4.0	0.76	16	Performing well- discontinued as a result of control failure
9735b	paste	60	70	15	4.0 6.0	0.83 0.77	4 16	performing well - discontinued as a result of control failure

FINANCIAL REPORT

For

NOVEMBER - 1962

(Contract NAS 3-2551)

Submitted by

NEW PRODUCT RESEARCH of TAPCO

(Attachment to Monthly Progress Report No. 5)

FINANCIAL REPORT

for

Contract NAS 3-2551

for

Period Ending November 30, 1962

	Current Month	Total To Date
TRW Engineering Hours	220	955.5
TRW Costs and Commitments	$ 3,300	$15,750
Subcontractor Costs and Commitments	18,500	61,660
TOTAL	$21,800	$77,410